	BACKGROUND OF THE INVENTION
2	This application is a continuation-in-part of
3	This application is a continuous filed February 6,
4	application US Serial No. 10/395,941, filed February 6,
5	2003.
6	This invention is directed to an additive/process
7	aid for polymeric materials such as rubber elastomers.
8	More particularly, this invention is directed to an
9	additive/process aid for such polymeric materials to
10	facilitate the processing and/or enhance the properties
11	of the elastomers.
12	Polymeric materials such as rubber elastomers are
13	used for the manufacture of various products including
14	tire components such as tire tread, wedge compounds,
15.	sidewalls, bead filler and tire carcasses. However, it
16	is well known that rubber elastomers tend to exhibit
17	certain undesirable physical characteristics, such as,
18	poor tear strength.
19	Tear strength or crescent tear is a measurement of
20	the resistance of a material to tear forces. The
21	higher the crescent tear measurement, the more
. 22	difficult it is for the elastomer to be torn from
23	whatever it is bonded to, such as steel.
24	Rubber elastomers also tend to exhibit weak
25	tensile strength. Tensile strength is a measurement of
26	the force at which a rubber elastomer can be expected
27	to fail under a tension load. The tensile strength of
28	the rubber elastomer is important in order to maintain
29	its elasticity. Specifically, for rubber elastomers
30	used in tires, the higher the tensile strength, then
31	reggire can be added to the tire, without
2.0	for of blowing the tire.

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Finally, rubber elastomers tend to have a low
1
    elastic limit or tan delta measurement. The tan delta
     measurement is a measurement of the stress and strain
 3
     exhibited on the elastomer or the amount of force that
 4
    can be applied before the elastomer reaches its elastic
 5
     limit and fails. It is advantageous for numerous
 6
     applications to increase the elastic limit of rubber.
          With particular regard to tire applications, it is
 8
     desirable to produce rubber elastomers or elastomeric
 9
    materials capable of exhibiting reduced hysteresis.
10
     Such rubber elastomers when fabricated into components
11
     for constructing articles such as tires, vibration
12
     isolators, power belts, and the like, will display
13
     properties of increased rebound, decreased rolling
14
     resistance and reduced heat-buildup when subjected to
15
16
     mechanical stress during normal use. Hysteresis refers
17
     to the difference between the energy applied to deform
     an article made from the elastomer and the energy
18
19
     released as the elastomer returns to its initial, un-
    deformed state. In pneumatic tires for instance,
20
     lowered hysteretic properties are associated with
21
     reduced rolling resistance and reduced heat build-up
22
     during operation of the tire. These properties, in-
23
24
     turn, result in lowered fuel consumption of vehicles
     using such tires and prolonged tire life. A reduction
25
     in the measured tan delta of the elastomer is
26
27
     indicative of a reduction in the hysteresis of the
28
     elastomer.
          To address these undesirable physical
29
30
     characteristics, process aids and/or additives were
     introduced into the rubber elastomer composition.
31
     Process aids are substances added to aid in the
32
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production processing of the rubber elastomers.

- Additives are substances added to improve specific 1
- physical characteristics of the rubber elastomer. 2
- Additives can also act as process aids and aid in the 3
- processing of the elastomers.
- Additives, such as calcium stearate and stearic 5
- acid, were used as process aids. However, the 6
- organic/organometallic nature of these substances 7
- causes them to have limited thermal stability and shelf 8
- life. Other prior process aids were products of 9
- organic chemical reactions. However, these process 10
- aids were not reproducible batch after batch and were 11
- often prone to error. Additives, such as carbon blacks 12
- and various silicas, were also added to improve, among 13
- other things, the tensile strength of rubber 14
- elastomers. 15
- Thus, there is a need for a process aid which 16
- overcomes the limitations of the prior additives, and 17
- is a simple, low-cost product that improves the 18
- physical characteristics of rubber elastomers by 19
- providing increased tensile strength, elasticity and 20
- tear strength. 21

SUMMARY OF THE INVENTION

- 22 In accordance with the present invention, there is 23
- provided an additive/process aid for polymeric 24
- materials such as rubber elastomers which overcomes the 25
- limitations of earlier additives/process aids.
- 26 Further, in accordance with the present invention,
- 27 there is provided an additive/process aid which
- 28 provides a simple, quick, and low-cost, slow speed
- 29 mixture blend which produces a stable product. It also 30
- improves the physical characteristics of rubber
- 31 elastomers, by providing, among other things, increased 32
- tensile strength, elasticity, and tear strength. 33

The additive/process aid comprises a mixture of 1 comminuted or micronized shale/slate predominately 2 formed of aluminum, magnesium and/or iron silicates, 3 starch, a reactive resin component, a metal carbonate and an acidic component such as a fatty acid or a 5 silica gel. 6. As used herein, comminuted shale/slate includes 7 gray slate flour (alumina silicate or ground shale) as 8 well as silicates of aluminum, magnesium and iron and mixtures thereof. The gray slate flour is typically in 10 powder form to assist in its dispersion in the 11 polymeric material. 12 Starch may be obtained from most agricultural 13 crops; the dominant starch raw materials being potato, 14 cassava, corn and wheat. Corn starch is preferred and 15 the present invention is described with particular 16 reference to the use of the same. 17 The resin component is selected from the group of 18 reactive polyesters resins and epoxy resins. 19 reactive cite of the polyester resin may comprise a 20 carbon-carbon unsaturation intermediate ester groups. 21 Preferred polyesters include those used in structural 2.2 applications such as reinforced laminates. The epoxy 23 resin may comprise a wide range of epoxies as described 24 below, but epoxidized soybean oil is preferred. 25 The metal carbonate preferably includes a metal 26 selected from Group IA or IIA of the Periodic Table. 27. Calcium carbonate is preferred and described below. 28 The acidic component may comprise silica gel or a 29 fatty acid. Useful fatty acids include stearic acid 30

and oleic acid.

The additive may be formulated with reduced 1 amounts of metal carbonate and increased amounts of the 2 remaining ingredients varying up to about: 30% flour, 3 35% starch, 15% resin, 5% metal carbonate and 15% 4 acidic component, by weight, wherein the aggregate 5 amount of the ingredients total 100%. On the other 6 hand, the metal carbonate may be increased and the 7 remaining ingredients reduced to about: 10% flour, 10% 8 starch, 5% resin, 70% metal carbonate and 5% acidic 9. component, by weight, wherein the aggregate amount of 10 the ingredients total 100%. Additive/process aids 11 having these "high" and "low" formulations have 12 provided improvements with acceptable trade-offs in 13 certain of the composition properties readily 14 identified by the skilled compounder. 15 In preferred compositions, the additive/processing 16 aid comprises from 10 to 30% shale/slate material, 10 17 to 35% starch, 5 to 15% resin, 10 to 40% metal 18 carbonate, 5 to 15% acidic component, by weight, 19 wherein the aggregate amount of the ingredients total 20 100%. More preferably, the additive/processing aid 21 comprises from 20 to 30% shale/slate, 20 to 35% starch, 22 5 to 10% resin, 20 to 35% metal carbonate, 8 to 15% 23 acidic component, by weight, wherein the aggregate 24 amount of the ingredients total 100%. 25 Preferred additive/processing aid compositions 26 including polyester resin and silica gel comprise from 27 20 to 29% gray slate flour, 18 to 28% corn starch, 4 to 28 8% liquid polyester, 10 to 20% calcium carbonate, 8 to 29 15% silica gel, by weight, wherein the aggregate amount 30 of the ingredients total 100%. More preferably, the 31 additive/processing aid contains about 29% gray slate 32 flour, about 28% corn starch, about 8% liquid 33

- 1 polyester, about 20% calcium carbonate, about 15%
- 2 silica gel, by weight, wherein the aggregate amount of
- 3 the ingredients total 100%.
- 4 Preferred additive/processing aid compositions
- 5 including epoxy resin and a fatty acid comprise from 20
- 6 to 30% gray slate flour, 20 to 35% corn starch, 5 to
- 7 10% epoxy, 10 to 20% calcium carbonate, 10 to 15% fatty
- 8 acid, by weight, wherein the aggregate amount of the
- 9 ingredients total 100%. More preferably, the
- 10 additive/processing aid contains about 25% gray slate
- 11 flour, about 25% corn starch, about 5% epoxy resin,
- 12 about 35% calcium carbonate, about 10% fatty acid, by
- 13 weight, wherein the aggregate amount of the ingredients
- 14 total 100%.
- As noted above, the resin may comprise a polyester
- 16 resin or an epoxy resin. The resins are preferably
- 17 liquid at rubber processing conditions and have a
- 18 molecular weight of from about 200 up to about 10,000
- 19 or higher.
- 20 Suitable epoxy compounds comprise molecules having
- 21 at least one or more epoxide group per molecule and
- 22 include epoxidized alkanes, alkenes, cycloalkanes,
- 23 alkenes and other epoxidized polymers and chemicals.
- 24 For example, epoxy/ether, epoxy/hydroxyl, epoxy/ester,
- 25 epoxy/amine, ether/amine, and cycloaliphatic
- 26 ether/hydroxyl group. These compounds contain
- 27 aliphatic and/or cycloaliphatic groups. Examples
- 28 include Octyl Epoxy Tallate (Argus Chemical and Union
- 29 Carbide), Epoxyprene 25 and 50 (Epoxidized Natural
- 30 Rubber from Guthrie Latex), Epon (Epoxidized Bisphenol
- 31 A from Shell Oil Co.), Styrene Oxide (S500-6 Aldrich
- 32 Chemical Co.) and 1,2-Epoxy-3-phenoxypropane (24,848-7
- 33 Aldrich Chemical Co.). Epoxidized plant(or vegetable)

	and the second s
'n	oils, such as epoxidized soybean oil © P Hall PARAPLEX
2	GC2 Argus Chemical, Union Carbide and Harwick
3	Standard) and epoxidized linseed oil (Argus Chemical),
4	are most preferred.
5	The additive/processing aid is preferably added to
6	polymeric materials such as rubber elastomers or
7	plastic materials such as polyethylene, ethylene
8	repulere The preferred rubber elastomers are
9	relogted from the group consisting of natural rubber,
10	isoprene, chloroprene, halo-butyls, SBR rubber, butyl
	rubber, neoprene, epichlorohydrin rubber,
11 12	polysulfonated rubber, silicone rubber and mixtures
13	thereof.
14	These and other aspects of the invention are
15	described below in further detail, the illustrated
16	embodiments being representative of only some of the
	ways in which the principles and concepts on the
17	invention can be executed and employed.
18	invention can be executed that I
	DETAILED DESCRIPTION OF
19	DETAILED DESCRIPTION OF PREFERRED AND ALTERNATE EMBODIMENTS
20	PREFERENCE Airported to an

The present invention is directed to an additive/process aid to facilitate the production processing of rubber elastomers. Once added to the rubber elastomers, the process aid tends to increase tensile strength, elasticity, and tear strength of these rubber elastomers.

The present invention is both an additive and a process aid. It is an additive because it is part of the final rubber product made by an end user. It is also considered a processing aid because the additive will aid dispersion and give the end user enhanced

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properties associated with homogeneity as well as more
1.
    through-put of product in a manufacturing process.
2
         Generally, it is desirable to prepare the
3
    additive/processing aid within the preferred ranges of
4
    ingredients. Deviations from these ranges may be
5
    readily determined by designed compound evaluations
                                          Although
    measured by the obtained properties.
    deviations from these ranges may occur with trade-off
8
    of various properties, the following observations have
    been made rubber elastomers and additive/process aids
10
     including gray slate flour, corn starch, liquid
11
     polyester, calcium carbonate and silica gel.
12
          Gray slate flour will typically be used in the
13
     range of between 20 and 30%. It is preferable to stay
14
     within this range since adding an excessive amount of
15
     gray slate flour may cause scorching of the
16
     additive/process aid and adding too little gray slate
17
     flour causes the additive/process aid to exhibit a low
18
      cure state or level of cure. Preferably, about 29 to
 19
      30% by weight of gray slate flour is added to the
 20
      additive/process aid. Gray slate flour helps to aid in
 21
      the reinforcement of the additive/processing aid.
 22
      Preferably, the gray slate flour has a particle size in
 23
      the range of 32 microns. Suitable commercially
 24
      available sources of gray slate flour include KeyStone
 25
      Corporation, sold under the name Gray Slate Flour.
 26
           Further, the additives/process aid should include
 27
      about 18 to 30% by weight of corn starch. Again, an
  28
      excess of corn starch tends to cause scorching of the
  29
       additive/process aid and a deficiency of corn starch
  30
       may cause the additive/process aid to exhibit a low
  31
       degree of cure. Preferably, about 28 to 30% by weight
  32
```

of corn starch is added to the additive/process aid.

- 1 Corn starch tends to increase the tear strength
- 2 enhancements of the additive/process aid. Preferably,
- 3 unhydrolyzed corn starch is added to the
- 4 additive/process aid. A suitable corn starch is
- 5 marketed by American Maze Corporation under the
- 6 designation Unhydrolyzed Corn Starch.
- 7 The additive/process aid also comprises at least
- 8 4% by weight and up to about 8% by weight of liquid
- 9 polyester. It is preferred to observe this range since
- 10 too much polyester tends to inhibit the incorporation
- of the additive/process aid into the rubber. On the
- other hand, if too little of the polyester is used,
- 13 scorching of the product tends to occur. Liquid
- polyester helps to aid in the reinforcement of the
- 15 additive/process aid. Preferably, the liquid polyester
- 16 is clear when added to the additive/process aid.
- 17 Suitable commercially available sources of liquid
- 18 polyester include Dynatron Corporation of Atlanta,
- 19 Georgia, the product being sold with the designation
- 20 Liquid Polyester Resin.
- 21 Calcium carbonate should be included in the
- 22 additive/process aid in accordance with the preferred
- ranges of from about 10% to about 35% by weight.
- 24 Permissive ranges of calcium carbonate vary from 5 to
- 25 70% by weight as indicated in the "high" and "low"
- 26 compositions described above. However, excessive
- 27 amounts of calcium carbonate may tend to cause
- 28 scorching of the additive/process aid and, too little
- 29 calcium carbonate, may result in a low degree of cure
- of the additive/process aid. Preferably, about 20% to
- 31 about 40% by weight of calcium carbonate is used, and
- more preferably, from about 32% to about 37% by weight
- 33 is used. Calcium carbonate improves the dispersion of

- 1 the additive/process aid. Preferably, calcium
- 2 carbonate is in a white form when added to the
- 3 additive/process aid. Calcium carbonate is available
- 4 from the Akrochem Company and from the J. Huber
- 5 Company, under the name Snowhite Calcium Carbonate.
- 6 The silica gel should also be used in the
- 7 preferred 8 to 15 % by weight range. Excess of amounts
- 8 of silica gel tend to cause scorching on the
- 9 additive/process aid and, too little silica gel, tends
- 10 to result in a low degree of cure. Preferably about
- 11 15% by weight of silica gel is used. Silica gel may be
- 12 added in the form of pellets or powder. Silica gel
- 13 helps to absorb water from the additive/process aid and
- 14 promotes more uniform dispersion. Preferably, the
- 15 silica gel is in a white form when added to the
- 16 additive/process aid. Silica gel may be obtained from
- 17 U.S. Silica Corporation under the name Minsil Silica
- 18 . Gel.
- The additive/process aid of the present invention
- 20 is prepared as a slow speed, mixer blend. The
- 21 components of the additive/process aid are added
- 22 together and mixed, and reacted under suitable time and
- 23 temperature conditions. Preferably, these components
- 24 are cured for 12 minutes at 307°F.
- The additive/process aid of the present invention
- 26 provides many advantages when added to a elastomeric
- 27 material such as a rubber elastomer, including improved
- 28 mixing, extrusion and processing characteristics, and
- 29 improved tear strength. It also maintains shore A
- 30 hardness and the cure state of the rubber elastomer.
- 31 Further, it increases tensile strength and elasticity
- 32 giving the rubber elastomer more flexibility. Finally,

it is a simple, low-cost additive/process aid, which 1 consistently produces a stable product. 2 The additive/process aid is preferably added to 3 rubber elastomers selected from the group consisting of: natural rubber, isoprene, chloroprene, halo-butyls, 5 SBR rubber, butyl rubber, neoprene, epichlorohydrin 6 rubber, polysulfonated rubber, silicone rubber and 7 The rubber elastomers find mixtures thereof. . 8 particular utility for tires, tire trends, carcasses 9 and sidewalls, coating stocks, hoses, belting, inner 10 tubes, inner liners, general-purpose rubbers and other 11 uses. 12 The additive/process aid is preferably added to 13 the rubber elastomer in the range of at least 1 phr and 14 up to 50 phr with consideration of the relative parts 15 of ingredients contained in the additive/process aid 16 Preferably, the additive/process aid is added 17 to the rubber elastomer in the range of from at least 2 18 phr and up to 30 phr. 19 The present invention is further exemplified in 20 the following examples which illustrate the use of the 21 additive/process aid in a typical rubber compounding 22 recipe. It is understood that the examples are only 23 illustrative of preferred embodiments according to the 24 present invention wherein the claims set forth the 25

27 <u>EXAMPLE A</u>

26

scope of the present invention.

The ASTM 297 Test Recipe for Truck Tread Tires was carried out, the resultant rubber elastomer from this test was prepared having the formulation shown in Table I.

1	TABLE I
2 3 4	RUBBER TEST RECIPE Parts per Hundred of Rubber
5 6 7 8 9 10 11 12 13 14	1. Natural Rubber (RSS or SMR-5) 53.6 2. cis-polybutadiene 36.0 3. napthenic process oil 2.0 4. stearic acid 5.0 5. zinc oxide 10.0 6. N-660 Black 2.0 7. nonox ZA 0.8 8. Santocure 2.0 9. Sulfur 10. Process Aid of Present Invention 30.0
15 16 17 18 19 20 21 22 23 24 25 26 27	
28	ther elastomer was prepared having the
29 30	attion shown in Table II.

1	TABLE II	
2 3 4	RUBBER TEST RECIPE Ingredients	Parts per Hundred of Rubber
5 6. 7 8 9 10 11 12 13	 Natural Rubber (RSS or SMR-5) cis-polybutadiene napthenic process oil stearic acid zinc oxide N-660 Black N-990 Black nonox ZA Santocure Sulfur 	36.0 2.0 5.0 20.0 35.0 2.0 0.8 2.0
15	The rubber elastomer composition	was again prepared as
16	described above, in Example A.	
17 18 19	COMPARATIVE EXAMPLE A rubber elastomer was prepformulation shown in Table III.	
20	TABLE .	III
21 22 23	RUBBER TEST RECIPE Ingredients	Parts per Hundred of Rubber
24 25 26 27 28 29 30 31 32 33	5. zinc oxide 6. N-660 Black 7. nonox ZA 8. Santocure 9. Sulfur	5) 53.6 13.4 36.0 2.0 5.0 20.0 2.0 0.8 2.0 55.0

- 34 The rubber elastomer composition was again prepared as
- 35 described above, in Example A.

_	Once these three rubber elastomer compositions
1	were prepared, laboratory tests were run, using ASTM
2	were prepared, laboratory tests were remark
. 3	297 test methods on each of the rubber elastomer
4	gempositions. The tests were designed to test specific
4 5	physical characteristics, such as tensile strength, 6
	elongation, and crescent tear of the rubber elastomer
6	compositions. The results of each of the tests
7	compositions. The results of caes of the clastomers are
8	performed on each of the Example rubber elastomers are
9	listed in the tables below.
	Tensile strength is a measure of the force at
10	which a rubber elastomer can be expected to fail under
11	which a rubber elastomer can be supplied the rubber
12	a tension load. The tensile strength of the rubber
13	olastomer is important in order to maintain its
	elasticity. It is often desirable that reinforcing
14	elasticity. It is offer and synthetic rubbers to
15	agents are added to natural and synthetic rubbers to
16	increase tear strength.

17	TABLE IV	
18 19	TENSILE STRENGTH Examples	psi
20 21 22	1. Example A 2. Comparative Example One 3. Comparative Example Two	2750 2500 2450

23 Percent elongation is a measurement of the 24 stiffness of rubber. The higher the number, the harder 25 the rubber composition to flux. It is often desirable 26 that to add reinforcing agents to natural and 27 synthetic rubbers to increase the percent elongation.

	-15-		
1	TABLE '	<u>V</u>	
2	PERCENT ELONGATION	· · · · · · · · · · · · · · · · · · ·	
3	Examples	percent	
		535%	
4	1. Example A 2. Comparative Example One	500%	
5	3. Comparative Example Two	489%	
6		landrogg of	
7	Shore A hardness is a meas	ure on the hardness of	
8	rubber. It also provides or in	dicates the cure state	
9		often desirable to and	
10	the reinforcing agents to natur	al and synthetic rubbers	
11	to maintain the Shore A hardnes	s.	
		•	
10	TABLE	<u>VI</u>	
12	TARDNIECC		
13	SHORE A HARDNESS Examples	Shore A hardness	
14	Examples	. 61	
15	1. Example A	61 61	
16	2. Comparative Example One	58	
17	3. Comparative Example Two		
18	Crescent tear or tear str	ength is the force	
19	necessary to propagate a crack	or tear under stress on	
20	a horizontal pull. It is ofte	n desirable to add	
21	reinforcing agents to natural	and synthetic rubbers to	
22	increase tear strength.		
	TABLE	VII	
23		•	
24	CRESCENT TEAR	Lb/inch	
25	Examples	•	
26	1. Example A	690	
27	2. Comparative Example One	650 632	
28	3. Comparative Example Two		
	Tan delta is a measure of the rolling resistance.		
29	Tan delta is a measure o	dalla value the lower the	
30	The lower the reading or tan	derica varue, che rondi	
31	rolling resistance. It is of	ten desirable to aud	
	7 - 3		

- 1 reinforcing agents to natural and synthetic rubbers to
- 2 decrease the tan delta.

3	TABLE V	III

-	TAN DELTA Examples	resistance
7	1. Example A 2. Comparative Example One 3. Comparative Example Two	0.162 0.179 0.192

- 9 The results of the rubber elastomer compositions
- 10 in Comparative Examples One and Two were compared to
- 11 Example A for changes in the physical properties
- 12 exhibited by the two rubber elastomer compositions. As
- 13 apparent from the above data, use of the
- 14 additive/process aid of the present invention (Example
- 15 A) resulted in significant increases in tensile
- 16 strength, percent elongation and crescent tear and
- 17 reduced tan delta measurements. Moreover, this rubber
- 18 elastomer composition also maintained its Shore A
- 19 hardness measurement. Accordingly, the
- 20 additive/process aid of the present invention imparts
- 21 favorable properties when utilized in association with
- 22 rubber elastomers.
- 23 Additionally, Example A contains the
- 24 additive/process aid of the present invention; it does
- 25 not contain large quanities of other additives/process
- 26 aids, such as silica or carbon black, as compared to
- 27 Comparative Examples One and Two. Thus, the improved
- 28 physical characteristics of the rubber elastomer
- 29 composition (Example A) are solely attributable to the
- 30 additive of the present invention.
- The additive/process aid including epoxidized oil
- 32 and fatty acid is illustrated hereinafter. In a

- 1 preferred formulation, the additive/process aid
- 2 includes the following ingredients.

ADDITIVE/PROCESS AID COMPOSITION

	_	ADDITIVE/PRO	CESS AID CO
	3	<u>NDD 1111</u>	
	4		DB6
	4	diont	(Wet. %)
	5	Ingredient	. 25
	6	gray slate flour	25
	7	corn starch	
	8	epoxidized soybean oil	5
	•	calcium carbonate	35
	9		1.0
-	10	stearic acid	10.

- 11 In accordance with the foregoing additive/process aid
- 12 formulation, a rubber elastomer composition Example B
- in accordance with the invention was prepared together
- 14 with a Comparative Example Three or Control 3 as
- 15 reported in the following Table IX.

TABLE	<u>IX</u>	
16 17 18 19 MATERIAL 20 Natural Rubber(SMR20)	EXAMPLE B PER 30.0 70.0	CONTROL 3 PER 30.0 70.0
Carbon Black(N339) DB6 Sunder 750 T Sunder 750 T Stearic Acid Zinc Oxide Akrowax 5030 Sulfur Accelerator CBS ¹ Co-accelerator DPG ²	50.0 2.0 10.0 2.0 2.0 2.5 2.0 2.0 1.7 0.2 174.4	50.0 0.0 10.0 2.0 2.5 2.0 2.0 1.7 0.2 172.4

- 33 34 ¹N-Cyclohexyl-benzothiazolesulfenamide
- 35 ²Diphenylguaidine
- 36 Mix Cycle: Example B and the Control were mixed in the
- 37 same manner except for the inclusion of DB6 in Example
- 38 B. Accordingly, the rubber and carbon black together

- 1 with DB6 in the case of Example B were mixed for 1.5
- 2 minutes at 60 RPM in a mixer preheated to 250°F. Oil
- 3 followed by Santoflex 134, stearic acid, zinc oxide and
- 4 Akrowax 5030 were added, and mixing continued to a
- 5 total mix time of 5.5 minutes. Then, the batches were
- 6 dumped, formed into sheets on a two roll mill and
- 7 allowed to cool.
- 8 Finish Cycle: The masterbatch followed by CBS, DPG and
- 9 sulfur are combined in a mixer preheated to 175°F and
- 10 mixed at 50 RPM for one minute or temperature increase
- 11 to 200°F, whichever came first. Each of the rubber
- 12 elastomers was then provided with 10 roll passes on a 2
- 13 roll mill followed by one cut pass.
- The compositions of Example B and Comparative
- 15 Example Three were tested and the results are reported
- 16 below in Table X.

1 77	TABI	LE X	
17		Example B	Control 3
18 19	Processability Testing	*	
20	Mooney Viscosity ML4@ 212°f	54	51
21 22	Monsanto Rheometer Minimum Torque, m-g	93.1	98.7
23 24 25	Maximum Torque, m-g Scorch, t1, min. 90% Cure Time min.	11.8 5.67 13.83	11.2 5.67 13.00
26	Physical Properties	·	
27 28 29 30 31 32 33	Stress-Strain Properties Room Temperature 23°C 50% Modulus, psi 100% Modulus, psi 200% Modulus, psi 300% Modulus, psi Tensile Strength, psi Elongation, %	285 573 1490 2539 3042 351	259 510 1351 2368 2928 357

. 1	TABLE X	Cont.	
2	Elevated Temperature 100°C		
3	50% Modulus, psi	275	244
	100% Modulus, psi	554	484
4	200% Modulus, psi	1301	1182
5	300% Modulus, psi	_	- .
6	Tensile Strength, psi	1404	1449
7	Elongation, %	219	228
, 8	Elongacion,		·
9	Shore A Hardness	7.0	70
10	Room Temperature 23°C	70	-
11	Elevated Temperature 100°C	_	
_ :	Tear Strength ASTM D624 Die C	*	
12	Room Temperature 23°C	193	190
13	Elevated Temperature 100°C	102	118
14	Elevated lemperature 100 0		
	Bashore Resilience, %	41	41
15	Bashore Restriction	62	60
16	Dynamic Properties 1 Hz		
17	Dylianic Flopergroup		
18	5% Compression		
19	Elastic Modulus, E'	0 0 - 1 0 6	7.60x10 ⁶
20	26°C	8.05x10 ⁶	6.33x10 ⁶
21	75°C	6.63x10 ⁶	5.85x10 ⁶
22	122°C	6.05x10 ⁶	5.85%10
23	Storage Modulus, E"		
	26°C	1.80x106	1.79×10^{6}
24	75°C	1.08x106	1.08×10^{6}
25	122°C	0.812×10^{6}	0.768×10^{6}
26	122 C		
27	Tangent	0 000	0.235
28	26°C	0.223	0.233
29	75°C	0.163	0.170
.30		0.134	0.131
31	10% Compression		
32	Elastic Modulus, E'		c ====1.06
33		6.81x10 ⁶	6.57x10 ⁶
3 <i>3</i>		6.04x10 ⁶	5.71x10 ⁶
35		5.77×10^{6}	5.39x10 ⁶
33	, <u> </u>		

	TARLE	X Cont.		
1 .		<u>A</u> COMO	•	
2 3 4 5	Storage Modulus, E" 26°C 75°C 123°C	1.39x10 ⁶ 0.876x10 ⁶ 0.687x10 ⁶	1.47x10 ⁶ 0.931x10 ⁶ 0.651x10 ⁶	
6 7 8 9	Tangent Delta 26°C 75°C 123°C	0.204 0.145 0.119	0.223 0.163 0.121	
10 11 12 13 14 15 16 17 18 19 20 21	The use of epoxy residularity illustrated in the following particularly, Examples C at the relative amounts or profession of the additive composition invention. More particular DB6H of Example C includes carbonate in favor of relative remaining ingredients and D includes a relatively his carbonate with reduced amount ingredients as summarized.	ng Examples C and D illustrate operations of the ns in accordance of the additional time and DB6L operamount of the results of the results of the results.	and D. More e variations in ne ingredients ce with the ive/process aid nt of calcium ounts of the used in Example calcium	
22	ADDITIVE APPOCESS AID COMPOSITIONS			
23 24	Ingredient	DB6H (Wet. %)	DB6L (Wet. %)	
25 . 26 27 28 29	corn starch epoxidized soybean oil calcium carbonate stearic acid	30 35 15 5 15	10 10 5 70 5	
30	Examples C and D in	accordance with	h the invention	
31	respectively include addi			
	the the additional constituents shown in			

DB6L together with the additional constituents shown in

the following Table IX. For comparison, a Control 4 or

32

- 1 Comparative Example Four was prepared as reported in
- 2 Table XI.

3	•	TABLE X	<u>I</u> .	
4 5		EXAMPLE C	EXAMPLE D PER	CONTROL 4 PER
6	MATERIAL			
7	Natural Rubber		20.0	30.0
8	(SMR20)	30.0	30.0	
9	SBR 1520	70.0	70.0	70.0
10	Carbon Black(N343)	41.0	41.0	41.0
11	DB6H	2.0	0.0	0.0
12	DB6L	0.0	6.0	0.0
13	Aromatic Oil(Shell	750) 10.0	10.0	10.0
14	Antioxidant		•	_
15	(Santoflex 13)	1.0	1.0	1.0
16	Stearic Acid	2.0	2.0	2.0
17	Zinc Oxide	2.5	2.5	2.5
	Akrowax 5030	1.0	1.0	1.0
18 19	Masterbatch Total	157.5	159.5	163.5
2.0	Masterbatch	157.5	159.5	163.5
20		1.3	1.3	1.3
21	Sulfur	1.7	1.7	1.7
22	Accelerator CBS ¹	_ :	0.2	0.2
23 24	Co-accelerator DPG Final Total	160.7	162.7	166.7

- 25 ¹N-Cyclohexyl-benzothiazolesulfenamide
- 26 ²Diphenylguaidine
- 27 Masterbatch Mixing procedure: Start temperature 250°F,
- 28 RPM'S 40, add pre-blended polymers and carbon black
- 29 (together with DB6H or DB6L in Examples B and C), 0
- 30 minutes, mix for 1.5 minutes. Add oil followed by
- 31 antioxidant, stearic acid, zinc oxide and wax, increase
- 32 RPM to 60. Mix for 4 minutes and dump. Sheet out and
- 33 cool.
- 34 Final Mixing Procedure: Start temperature 175°F, RPM's
- 35 50. Add masterbatch, followed by remaining
- 36 ingredients. Mix keeping temperature below 200°F.
- 37 Dropped and performed 10 cut passes and 10 roll passes
- 38 on mill.

Examples C and D together with the Control 4 or
Comparative Example Four were cured for 15 minutes at
149°C and tested. The results of the tests are
reported below in Table XII.

5		TABLE XII	•	
		Example C	Example D	Control 4
6		2	-	
7	Rheometrics @ 149°C	TO 60	75 26	73.94
8	MH	72.60	75.36	11.13
9	ML	10.52	11.74	13.93
10	TC80	14.89	13.43	16.77
11	TC90	18.13	16.75	7.98
12	TS2	8.32	7.11	9.24
13	TS5	9.63	8.20	9.24
14	Mooney Viscosity	•		
15	ML(1+4)@100°C	40.31	45.19	40.30
1.0	Shore A Hardness			
16	Hardness@23°C	55.2	55.0	55.2
17	Hardness@100°C	66.2	67.4	66.0
18	naturesservo c			
19	Pendulum Rebound	F2 7	56.3	53.2
20	Rebound@23°C	52.7	52.8	48.8
21	Rebound@100°C	49.6	52.0	10.0
22	Stress-Strain Propertie	es		
23	Results at 23@°C			100
24	100% Modulus, psi	192	235	183
25	200% Modulus, psi	474	618	451
26	300% Modulus, psi	1031	1314	956
27	Tensile Str., psi	2814	2962	2317
28	Elongation, %	534	496	500
29	Results at 100@°C	176	211	170
30	100% Modulus, psi	401	504	385
31	200% Modulus, psi	801	984	754
32	300% Modulus, psi	1225	1247	1182
33	Tensile Str., psi	392	351	402
34	Elongation, %	392	331	
35	Tear Strength		407 4	462 4
36	Pounds/in.@23°C	556.9	427.4	463.4
37	Pounds/in.@100°C	310.7	241.6	336.4
3.8	Specific Gravity	1.0746	1.0659	1.0686

TABLE XII Cont. 1 Tangent Delta1 2 0.13385 0.15172 0.14305 Tan Delta@ 5% strain 3 % decrease from Control 5.714 11.778 na 4 0.14654 0.12925 Tan Delta@ 10% strain 0.13876 5 % decrease from Control 5.309 11.799 na 6 1(50°C, 1HZ, Strain Sweep) 7 As indicated by the foregoing comparisons, the 8 additive/process aid in accordance with the present 9 invention imparts increased tensile strength especially 10 under conditions of elevated temperature and high 11 In addition, hysteresis is reduced as 12 indicated by the decrease in the tan delta values as 13 compared with the Control or Comparative Example Three. 14 Reduced hysteresis is associated with reduced rolling 15 resistance and reduced heat build-up during tire 16 operation or other cycled loading of the rubber 17 elastomer which give rise to lower fuel consumption 18 and/or increased elastomer product life. 19 Although preferred embodiments have been described 20 in detail, it should be understood that various 21 changes, substitutions, and alterations can be made 22 therein without departing from the spirit and scope of 23 the invention as defined by the appended claims. 24 will be appreciated that various changes in the 25 details, materials and arrangements of parts, which 26 have been herein described and illustrated in order to 27 explain the nature of the invention, may be made by 28 those skilled in the area within the principle and 29 scope of the invention as will be expressed in the 30

appended claims.